फेरोटाइटेनियम का रासायनिक विशलेषण

(पहला पुनरीक्षण)

Methods of Chemical Analysis of Ferrotitanium

(First Revision)

ICS 77.100

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April 2019 **Price Group 6**

Methods of Chemical Analysis Sectional Committee, MTD 34

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The earlier Indian Standard on methods of chemical analysis of ferrotitanium were covered by the following parts:

IS No.	*Title*
13840	Chemical analysis of ferrotitanium
(Part 1) : 1993	Determination of carbon by direct combustion gravimetric method
(Part 2) : 1993	Determination of silicon by gravimetric method
(Part 3) : 1993	Determination of titanium by cupferron (gravimetric) method
(Part 4) : 1998	Determination of aluminium by phosphate method
(Part 5) : 1998	Determinations of sulphur by direct combustion method
(Part 6) : 1998	Determination of titanium by x-ray fluorescence technique

The Committee while reviewing this standard decided to have a single standard by merging all the above six parts of IS 13840. With the publication of this standard all the six parts of IS 13840 will be superseded. In this revision an alternate method has been added for determination of titanium content in ferrotitanium by titrimetric method.

In the formulation of this standard considerable assistance have been derived from the ISO 7692 : 1983 'Determination of titanium content in ferrotitanium by titrimetric method' issued by International Organization of Standardization (ISO).

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF FERROTITANIUM

(First Revision)

1 SCOPE

This standard describes the method of chemical analysis of ferrotitanium.

2 REFERENCES

The standards listed below contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision)*
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
266 : 1993	Sulphuric acid — Specification (*third revision)*
1070 : 1992	Laboratory grade water (*third revision*)
1472 : 1977	Methods for sampling of ferro-alloys for determination of chemical composition (*first revision*)
6226 (Part 1) : 1994	Recommendation of apparatus for chemical analysis of metals: Part 1 Determination of carbon by direct combustion (*first revision*)
(Part 2) : 1987	Determination of sulfur by direct combustion

3 SAMPLING

3.1 The sample shall be drawn and prepared as per the method prescribed in IS 1472.

3.2 The sample is to be cleaned with analytical grade ether and acetone, dried in an air oven at 100 ± 5°C.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF CARBON UP TO EIGHT PERCENT BY THE DIRECT COMBUSTION GRAVIMETRIC METHOD

5.1 Outline of the Method

The sample is burnt in a stream of purified oxygen and the carbon dioxide formed is absorbed, after purification, in suitable absorbent and determined gravimetrically.

5.2 Reagents

5.2.1 *Oxygen*, Purity more than 99.5 percent

5.2.2 *Ascarite or Soda Lime*, Size 0.80 to 2.00 mm, dia - 20 to 30 mesh

5.2.3 *Magnesium Perchlorate* $Mg(ClO_4)_2$, Anhydrous

5.2.4 *Boat/Crucible*

Boat/crucible of precise dimensions for accommodating in the resistance/induction furnace.

5.2.4.1 Pre-ignite the boats/crucibles in air or oxygen in a furnace for an hour at 1 100 °C and store in a desiccator and check for consistency of the blank value.

5.2.5 *Flux/Accelerator*

Copper, red lead (pre-ignited at 550°C), tin and iron of low carbon content. Magnesium perchlorate.

5.3 Apparatus

5.3.1 The apparatus recommended IS 6226 (Part 1), may be used.

5.3.2 Instead of the resistance furnace an induction furnace may also be used.

5.4 Procedure

5.4.1 Assemble the apparatus. Switch on the furnace, if it is a resistance furnace, and allow it to attain a temperature of 1250°C to 1350°C, while passing oxygen through the apparatus so that it bubbles freely at the exit end of the train.

Disconnect the absorption bulb, keep in a desiccator till it attains room temperature and take the initial weight. Repeat the operation till a constant mass is obtained.

5.4.2 Weigh to the nearest 0.001 g, 2.0 to 10.0 g for carbon content up to 3 percent and 0.2 g for carbon content above 3 percent of the test sample. Transfer to the pre-ignited combustion boat covered at the bottom with a thin layer of calcined alumina spread the sample evenly over the top of the alumina and cover it with 2.0 to 8.0 g of the flux. Introduce the boat slowly in the hot zone of the combustion tube.

5.4.3 In the case of induction heating, weigh to the nearest 0.001 g, 0.9 to 1.1 g of the sample and transfer to a pre-ignited crucible. Add an equal quantity of the flux. Place to crucible in position on the pedestal post of the furnace, raise to the combustion position and lock the system. Pass oxygen through the system and ignite the sample.

5.4.4 Maintain a rapid flow of oxygen (800 to 1 000 ml/min) throughout the combustion, then reduce to 400 to 500 ml/min and maintain it for another 6 to 8 min in order to sweep out the carbon dioxide.

5.4.5 Remove' the absorption bulb and weigh it after keeping it in desiccator till it attains room temperature. The increase in mass of the bulb represents the mass of carbon dioxide.

5.4.6 Remove the boat or crucible and examine for any incomplete combustion. If the sample is not thoroughly fused, repeat the determination with a fresh sample.

5.4.7 Make a blank determination following procedure specified in **5.4.2** to **5.4.6** and using same quantity of reagents except the sample.

5.5 CALCULATION

$$\text{Carbon, percent by mass} = \frac{A - B}{C} \times 27.29$$

where,

A = increase of mass of the absorption bulb from the sample, in g;

B = increase of mass of the absorption bulb from the blank determination, in g; and

C = mass of the sample taken, in g.

6 DETERMINATION OF SILICON UP TO TWENTY FIVE PERCENT BY GRAVIMETRIC METHOD

6.1 Outline of the Method

The sample is decomposed by fusion with sodium peroxide. Silica is separated by dehydration from the acidified extract after addition of oxalic acid and finally determined gravimetrically.

6.2 Reagents

6.2.1 *Sodium Peroxide,* Solid

6.2.2 *Oxalic Acid Solution* 100 (g/l)

Dissolve 100 g of oxalic acid in water and dilute to one litre.

6.2.3 *Hydrofluoric Acid*, 40 percent

6.2.4 *Dilute Sulphuric Acid*, 1 : 1, 1 : 200 (*v/v*)

6.3 Procedure

6.3.1 Weigh 0.500 g of finely ground dried sample for silicon content up to 15 percent and 0.25 g of sample for silicon above 25 percent range, into a nickel crucible containing 4 to 8 g of sodium peroxide (*see* **6.2.1**). Mix well. Fuse the content over the dull flame of Mecker burner or in a muffle furnace at a temperature of 700°C to 800°C. Care should be taken not to raise the temperature rapidly, as to cause spattering. When the mass is molten, a slight rotary motion is imparted to the crucible, to stir up any unattacked particles of alloy, on the bottom or sides.

The temperature is to be increased to a bright redness for a minute just before completion of fusion. If followed carefully a very normal fusion without any spattering will result. When the crucible has cooled and the fusion has solidified, slowly drop the crucible in 50 ml of cold water in a 500 ml Polytetrafluoroethylene (PTFE) beaker and cover. When the reaction has subsided, take out the crucible and wash with dilute sulphuric acid (1 : 1) (*see* **6.2.4**) and then with water, collecting all the washings in the same PTFE beaker.

6.3.2 Neutralize the extract (*see* **6.3.1**) with dilute sulphuric acid (1 : 1) and add 50 ml of dilute sulphuric acid (1 : 1) and then transfer the solution to 500 ml glass beaker. Add 50 ml of oxalic acid solution and evaporate slowly to fumes. Heat to dense white fumes and continue the fuming for 3 to 5 min. Cool, add 200 ml of warm water and heat to dissolve sulphates. Filter through medium textured filter paper and wash at least 20 times with hot dilute sulphuric acid (1 : 200) and finally with hot water.

6.3.3 Transfer the residues along with the filter paper to a platinum crucible, Heat to dryness. Carefully char the filter paper and finally ignite at 1 100°C to constant mass. Allow to cool in a desiccator and weigh the crucible containing impure silica (*A*).

6.3.4 Moisten the impure silica with few drops of water and add about 2 or 3 drops of sulphuric acid. Add about 5 ml of hydrofluoric acid and evaporate to dryness until fumes cease to evolve. Ignite in a muffle furnace at 1 100 °C to a constant mass. Allow to cool in a desiccator and weigh the crucible (*B*).

6.3.5 *Blank*

Carry out a blank determination following the procedure specified in **6.3.1** to **6.3.4** and using same amount of all reagents. Correct the final results for the blank value, if any (*C*).

6.4 Calculation

$$\text{Silicon, percent by mass} = \frac{[(A - B) - C\,] \times 46.74}{D}$$

where,

A = mass of crucible before hydrofluorization, in g;
B = mass of crucible after hydrofluorization, in g;
C = mass of silica from blank, in g; and
D = mass of sample taken, in g.

7 DETERMINATION OF TITANIUM FROM TWENTY TO SEVENTYFIVE PERCENT BY CUPFERRON (GRAVIIMETRIC) METHOD

7.1 Outline of the Method

The sample is dissolved in sulphuric acid, nitric acid and hydrofluoric acid. Hydrogen sulphide separations are made in acid and alkali tartarate media. Titanium is then precipitated with cupferron, ignited and weighed as oxide. Corrections are applied for vanadium and zirconium, if present.

7.2 Reagents

7.2.1 *Dilute Sulphuric Acid,* 1 : 1, 1 : 4 and 1 : 9 (*v/v*)

7.2.2 *Hydrofluoric Acid,* 40 percent

7.2.3 *Nitric Acid,* rd = 1.42 (*see* 1S 264)

7.2.4 *Tartaric Acid Solution, (*500 *g/l*)

Dissolve 500 g of tartaric acid in water and make up to one litre

7.2.5 *Hydrogen Sulphide,* Gas

7.2.6 *Hydrogen Sulphide Wash Solution*

Add 20 ml of dilute sulphuric acid (1 : 1) to 20 ml of tartaric acid solution (*see* **7.2.4***)* and dilute the solution to one litre. Saturate it with hydrogen sulphide gas.

7.2.7 *Ammonium Hydroxide,* rd = 0.90

7.2.8 *Ammonium Sulphide Wash Solution*

Dissolve 20 g of ammonium chloride and 10 g of ammonium tartarate in 500 ml of water. Add ammonium hydroxide in slight excess, dilute to one litre and saturate with hydrogen sulphide gas.

7.2.9 *Cupferron Solution,* 6 percent

Dissolve 6 g of cupferron in 80 ml of cold water dilute to 100 ml and filter Prepare fresh as required.

7.2.10 *Cupferron Wash Solution*

Dilute 20 ml of cupferron solution (*see* **7.2.9**) to one litre with cold dilute sulphuric acid (1 : 9).

7.2.11 *Potassium Bisulphate,* Solid

7.2.12 *Hydrogen Peroxide*, 20 volumes

7.2.13 *Ammonium Phosphate Solution,* 10 percent

Dissolve 100 g of diammonium hydrogen phosphate in water and dilute to one litre. Filter before use.

7.2.14 *Sulphuric Acid,* rd = 1.84 (*see* IS 266)

7.2.15 *Perchloric Acid*, 70 percent (*v/v*)

7.2.16 *Phosphoric Acid*, 85 percent (*v/v*)

7.2.17 *Ammonium Nitrate Solution,* 50 (*g/l*)

Dissolve 50 g of ammonium nitrate in water and dilute to 1 litre.

7.2.18 *Iron Standard Solution* (*1 ml = 0.01 mg Fe*)

Transfer 0.20 g of iron (purity 99.8 percent) to a 250 ml beaker. Add 10 ml hydrochloric acid (1 : 1) and 1 ml bromine water. After dissolution add 10 ml of sulphuric acid and heat to copious fume. Cool, dilute and make up to 200 ml.

7.2.19 *Titanium Standard Solution* (*1 ml = 0.10 mg Ti*)

Fuse 0.17 g of titanium dioxide in a covered platinum crucible with 15 g of potassium bisulphate. Cool the melt and dissolve in 200 ml of hot dilute sulphuric acid (1 : 1) stirring occasionally. Cool, transfer to a 1 000 ml volumetric flask. Dilute to volume and mix.

7.3 Procedure

7.3.1 Weigh 0.500 g of sample and transfer to a 100 ml platinum dish. Cover and add 5 ml of dilute sulphuric acid (1 : 1), 5 ml of hydrofluoric acid (*see* Note) and sufficient nitric acid dropwise to effect dissolution. Remove and rinse the cover and sides of the dish.

Evaporate to dryness and cool. Add 30 ml of dilute sulphuric acid (1 : 4) stir well, heat for a few minutes and transfer to a 400 ml beaker. Heat until all sulphates have dissolved. Dilute to 200 ml, add 20 ml of tartaric acid solution and pass a brisk current of hydrogen sulphide for at least 20 min.

NOTE — 10 ml of the hydrofluoric acid shall be taken, if silicon is over 10 percent.

7.3.2 If a precipitate forms, filter through a paper containing paper pulp, into a 600 ml beaker. Wash about 20 times with hydrogen sulphide wash solution. Take the filtrate or take the clear solution if filtration was required, add paper pulp and make alkaline by adding ammonium hydroxide in slight excess. Pass a brisk current of hydrogen sulphide through the solution for at least 7 min and filter as soon as the precipitate settles, through a medium textured filter paper containing paper pulp, into a 800 ml beaker. Wash the paper and the precipitate about 20 times with ammonium sulphide wash solution (*see* **7.2.8**) preserve the paper and the precipitate.

7.3.3 Boil the filtrate for about 10 min then add 25 ml of dilute sulphuric acid (1 : 1) and boil down to 150 ml. Cool at 15 to 20°C, then add a little paper pulp and a slight excess of cold, freshly prepared cupferron solution (*see* **7.2.9**). Stir well and filter through two superimposed medium textured filter papers containing a little paper pulp and supported in a Buchner funnel using moderate suction. Wash about 20 times with cold cupferron wash solution (*see* **7.2.10**).

7.3.4 Ignite the paper and the precipitate in a weighed platinum dish, first at a low temperature to burn off carbonaceous matter and finally at 1 050 °C to 1 150°C for 20 min. Cool and weigh as oxides of titanium with other impurities. The ignited residue may contain oxides of vanadium and zirconium and therefore their mass shall be deducted from the mass of impure titanium dioxide.

7.3.4.1 *Estimation of zirconium dioxide* (ZrO_2)

Fuse the oxides (*see* **7.3.4**) with about 5 g of potassium bisulphate (*see* **7.2.11**) and dissolve the cold melt in 70 ml of dilute sulphuric acid (1 : 9). Add 5 ml of hydrogen peroxide (*see* **7.2.12**) and 25 ml of filtered diammonium hydrogen phosphate and allow to and overnight at a temperature of 70°C, stirring occasionally during precipitation.

Filter through a medium textured filter paper, containing paper pulp. Wash about 20 times with dilute hydrochloric acid (2 : 98) and finally about 5 times with ammonium nitrate solution (*see* **7.2.17**). Ignite in a weighed platinum crucible, first at a temperature not over 600°C until the carbonaceous matter has burnt off, and finally at 1 050°C to 1 100°C to a constant mass. Cool and weigh as zirconium pyrophosphate (ZrP_2O_7). Calculate zirconium dioxide (ZrO_2) as follows:

$$ZrP_2O_7 \text{ X } 0.464\,7 = ZrO_2 \qquad \text{------ (1)}$$

7.3.4.2 *Estimation of vanadium trioxide* (V_2O_3)

Weigh accurately 1 g of the ferrotitanium sample in a platinum dish. Treat with 20 ml of dilute sulphuric acid (1 : 1) (*see* **7.1.1**), 5 ml of hydrofluoric acid (*see* **7.2.2**) and sufficient nitric acid (*see* **7.2.3**) dropwise to effect dissolution of sample. Evaporate until dense white fumes just begin to evolve, cool and rinse into a 600 ml beaker containing 25 ml of boric acid solution. Dilute to 200 ml, add 40 ml of nitric acid and boil for 1 h or longer so that the final volume will be 100 to 125 ml. Dilute to 300 ml with cold water. Cool to 10°C and titrate potentiometrically with 0.20 N ferrous ammonium sulphate solution. Calculate any vanadium found as *vanadium trioxide* (V_2O_3) as follows:

$$V \text{ X } 1.785\,1 = V_2O_3 \qquad \text{------- (2)}$$

7.3.5 Place the paper and the precipitate of ferrous sulphide reserved under **7.3.2** to the beaker. Add 25 ml of nitric acid (*see* **7.2.3**) and 10 ml each of sulphuric acid (*see* **7.2.14**) and perchloric acid (*see* **7.2.15**). Evaporate to dense white fumes to destroy all organic matter. Add 100 ml of water and digest until salts are in solution. Transfer to a 200 ml volumetric flask, dilute to the mark and mix thoroughly.

Transfer a portion of the solution to a blank cell of a spectrophotometer. To the remaining solution in the volumetric flask add 1 ml of hydrogen peroxide (*see* **7.2.12**) and mix thoroughly. Fill the other cell with the solution. Measure the absorbance against the blank at 425 nm. Read the amount of titanium from the calibration curve.

7.3.6 *Calibration Curve*

Transfer 0, 2, 5, 10 and 20 ml of standard titanium solution (1 ml = 0.1 mg Ti) to 200 ml volumetric flask. Add 15 ml of standard iron solution (*see* **7.2.18**) and 80 ml of sulphuric acid (1 : 9) (*see* **7.2.1**). Add 1 ml of hydrogen peroxide (*see* **7.2.12**) and make up to the mark. Mix thoroughly. Measure the absorbance of the solution, against the first mentioned solution as 425 nm. Plot the absorbance readings against mg of titanium per 200 ml of solution.

7.4 Calculation

$$\text{Titanium, percent by mass} = \frac{(A \text{ x } 0.599\,5) + B}{C} \text{ x } 100$$

where,

A = mass of titanium dioxide corrected for ZrO_2 and V_2O_3, in g;

B = mass of titanium from ferrous sulphide precipitate (**7.3.5**), in g; and

C = mass of the sample taken, in g.

8 DETERMINATION OF ALUMINIUM FROM ONE TO TEN PERCENT BY CUPFERRON (GRAVIMETRIC) METHOD

8.1 Outline of the Method

Sample is dissolved in acid mixture, and after cupferron separation, aluminum is precipitated, filtered and ignited as oxide and weighed.

8.2 Reagents

8.2.1 *Dilute Sulphuric Acid,* 1 : 1 and 1 : 9 *(v/v)*

8.2.2 *Hydrofluoric Acid*

8.2.3 *Nitric Acid, rd=1.42* (conforming to IS 264)

8.2.4 *Dilute Hydrochloric Acid,* 1 : 1 *(v/v)*

8.2.5 C*upferron Solution,* 6 *percent*

Weigh 6 g of cupferron and dissolve in 80 ml of cold water. Dilute to 100 ml, filter and prepare fresh.

8.2.6 *Cupferron Wash Solution*

Dilute 20 ml of cupferron solution *(see* **5.2.5**) to 1 litre of cold dilute sulphuric acid (1 : 9).

8.2.7 *Perchloric Acid,* 70 percent

8.2.8 *Ammonium Chloride,* solid

8.2.9 *Bromocresol Purple,* 0.2 g/50 ml water

8.2.10 *Ammonium Hydroxide Solution,* 1 : 1 (*v/v*)

8.2.11 *Ammonium Chloride Wash Solution,* 2 percent.

8.3 Procedure

8.3.1 Weigh accurately 0.5 g of finely powdered sample and transfer to a 100 ml platinum dish. Add 20 ml of dilute sulphuric acid (*see* **8.2.1**) (1 : 1) and 5 ml of hydrofluoric acid (*see* **8.2.2**). Add nitric acid (*see* **8.2.3**) drop by drop to effect dissolution. Cover and warm till reaction is over. Remove from hot plate, wash the cover and sides of the dish and evaporate to fumes at low temperature. Cool and add 50 ml of dilute sulphuric acid (*see* **8.2.1**) (1 : 9) or 50 ml of dilute hydrochloric acid (1 : 1) (*see* **8.2.4**) and digest to dissolve salts. Transfer to 400 ml beaker, rinse and wash the dish. Cool below 5 to 10°C and add little paper pulp. Slowly add freshly prepared cupferron solution (*see* **8.2.5**) till the precipitation is complete and a little excess as seen by white colour of precipitate and stir vigorously for 2 to 3 min. Filter through medium texture filter paper. Wash thoroughly with cupferron wash solution (*see* **8.2.6**). Reserve the filtrate.

8.3.2 Return the precipitate to the original beaker and add 10 ml of sulphuric acid and 50 ml of nitric acid and evaporate to fumes. Add more nitric acid, if necessary, and repeat the evaporation to oxidize all organic matter. Cool, add 100 ml of water and digest until all salts are in solution. Cool below 20 °C and reprecipitate with cupferron solution, filter and wash as in **8.3.1**. Combine this filtrate with that reserved in **8.3.1**.

8.3.3 To the combined filtrate add 40 ml of nitric acid and 20 ml of perchloric acid. Evaporate to fumes and remove all organic matters. If organic matter still remains, add 10 ml more of perchloric acid and fume again. Cool, dilute to 200 ml with water and add 5 ml of dilute hydrochloric acid (1 : 1). Boil for 5 min and filter. Wash with water. Add 10 g of ammonium chloride (*see* **8.2.8**), 5 drops of bromocresol purple indicator and ammonium hydroxide solution (1 : 1) till the colour changes to purple (*see* **8.2.9**). Add paper pulp and boil for 1 min. Filter through a medium texture filter paper and wash thoroughly with ammonium chloride wash solution (2 percent) (*see* **8.2.11**).

8.3.4 Transfer the precipitate with paper to a beaker. Redissolve in 100 ml of dilute hydrochloric acid (1 : 1) and add 10 g of ammonium chloride. Boil and carry out the precipitation and filtration, followed by washing as before. Dissolve precipitate in 50 ml Hydrochloric acid (1 : 3). Wash filters with hot water. To the filtrate, add 0.5 g Ammonium bi phosphate $\{(NH_4)_2\ HPO_4\}$, a little pulp and two drops of methyl red (0.4 g/l). Make first ammoniacal, restore pink colour by adding Hydrochloric acid (1 : 19) drop by drop. Heat to boil add 20 ml ammonium acetate (200 g/l). Continue boiling for 5 min, allow the precipitate for settle. Filter, wash with hot ammonium nitrate (50 g/l) till free of chloride. Ignite in a platinum/porcelain, crucible cover, heat at 1 000 °C to constant weight. Weigh as aluminum phosphate.

8.3.5 Ignite the precipitate slowly, in a platinum crucible and then at 1 100°C to constant mass. Cool in a desiccator and weigh.

8.4 Calculation

$$\text{Aluminium, percent by mass} = \frac{(A\text{-}B) \times 0.228 \times 100}{C}$$

where,

A = weight of aluminium phosphate,

B = weight of blank solution, and

C = weight of sample.

8.4.1 Precision has to be established by analysing same sample in different laboratories. Expected + 0.15 at 5 percent level.

9 DETERMINATION OF SULPHUR FROM 0.005 TO 0.2 PERCENT BY DIRECT COMBUSTION METHOD

9.1 Outline of the Method

The sulphur in the sample is converted to sulfur dioxide by combustion in stream of oxygen in a furnace at 1 000°C. It is then absorbed in hydrochloric acid solution and titrated with standard potassium iodate solution, using starch as indicator.

9.2 Reagents

9.2.1 *Dilute Hydrochloric,* 3 percent (*v/v*)

9.2.2 *Starch Solution*

Transfer 1.00 g of pure starch powder and 2 ml of water in a small beaker and stir until a smooth paste is obtained. Pour this mixture into 50 ml of boiling water and cool. Add 1.5 g potassium iodide and stir until dissolved. Dilute to 100 ml. This solution is to be prepared afresh every time.

9.2.3 *Oxygen,* Purity *99.5* percent minimum

9.2.4 *Standard Potassium Iodate Solution,* (1ml = 0.1 mg S)

Dissolve 0.222 5 g of potassium iodate in 900 ml of water and dilute to 1 litre.

9.2.4.1 *Standard potassium iodate solution,* (1 ml = 0.02 mg S)

Transfer 10 ml of standard potassium iodate solution (*see* **9.2.4**) to a 500 ml volumetric flask, make up with water and mix well.

NOTE — However an empirical factor must be determined by analysing ECRM 548-1 (S = 0.030 percent) standard sample or any other certified sample.

9.2.5 *Standard Sample*

It should be of known sulphur content in the range of analysis.

9.2.6 C*eramic Boats/Crucibles*

Pre-ignited at 1 000 °C and stored in a desiccator.

9.2.7 *Accelerators*

Iron, tin, etc, free from sulphur.

9.3 Apparatus

The combustion furnace and titration unit as described in IS 6226 (Part 2) is most suitable. However, an induction furnace may also be used with similar purification and titration unit.

9.4 Procedure

9.4.1 Arrange the apparatus as given in IS 6226 (Part 2).

9.4.2 Switch on the furnace with oxygen bubbling through absorption vessel, which is previously filled with dilute hydrochloric acid (3 percent) (*see* **9.2.1**). Add 2 ml of starch solution (*see* **9.2.2**) to the vessel. Adjust the flow of oxygen from 1.0 to 1.5 l/min. Fill the burette with potassium iodate solution and add it to the absorption solution dropwise till a blue colour is obtained.

9.4.3 Weigh 1 g of the standard sample and transfer it into a pre-ignited boat/crucible. Add sufficient amount of accelerators (1 g of iron chips and 1 g of tin granules). After the unit has attained required warming that is about 30 min. introduce the boat/crucible with standard sample and accelerators in the furnace. Close the furnace. Burn the sample while passing oxygen all the time. Titrate continuously with potassium iodate solution at such a rate so as to maintain as close as possible to the original intensity of the blue colour.

NOTES

1 For low sulphur content, the standard potassium solution given in **9.2.4** can be used.

2 The end point can be detected, if the blue colour is stable for 1 min without further addition of potassium iodate solution. Record the volume in ml of potassium iodate used.

9.4.4 Refill the titration vessel with dilute hydrochloric acid (3 percent). Add 2 ml of starch solution and follow the procedure specified in **9.4.3** using test sample. Titrate the absorption solution with potassium iodate to the preselected end point and record the volume in ml of potassium iodate used *(A)*.

9.4.5 Determine the blank by placing the same amount of accelerator used in test sample in pre-ignited crucible and following the same procedure as specified in **9.4.3**. Deduct the blank value *(B)*. if any from the final calculation.

9.5 Calculation

$$\text{Sulphur, percent by mass} = \frac{(A - B)}{C} \times \frac{F}{10}$$

where,

A = volume of potassium iodate solution required for titration of the sample, in ml;

B = volume of potassium iodate solution required for the titration of blank, in ml;

C = mass of standard sample, in g; and

F = factor equivalent to sulphur for standard solution for potassium iodate, in mg.

10 DETERMINATION OF TITANIUM FROM TWENTY TO SEVENTY FIVE PERCENT BY X-RAY FLUORESCENCE SPECTROMETRIC METHOD (ALTERNATE METHOD)

10.1 Outline of the Method

The sample of ferrotitanium is fused with lithium metaborate and made into solid button. The plain surface of the button is exposed to primary X-rays. The secondary fluorescent radiation from the sample is dispersed and detected. The concentration, of titanium is obtained by relating to the titanium fluorescent X-ray intensity from the unknown sample to that of the standard reference material of known composition.

10.2 Equipments and Accessories

The different equipments that are required are pulverizer, crucibles and casting dish/moulds, muffle furnace and wavelength dispersive XRF spectrometer.

10.2.1 *Pulverizer*

It consists of a grinding box that can be set into vibrations of high frequency so that the pieces of samples placed in it can be powdered to 200 mesh size in 3 to 4 min.

10.2.2 *Crucibles and Casting Dish Moulds*

The crucibles and casting dish moulds made of a special alloy of 85 percent Platinum (Pt), 10 percent Rhodium (Rh) and 5 percent gold (Au) may be used for the preparation of beads.

10.2.3 *Muffle Furnace*

An electric furnace capable of achieving a temperature of 1 200 °C for a desired period of time must be used.

10.2.4 *Wavelength Dispersive X-ray Fluorescence Spectrometer*

It consists of an X-ray tube, a spectrometer chamber, a sample chamber, a mechanical arm, monochromators, a computer and associated electronic circuits. The X-ray tube acts as a source of primary X-rays. The spectrometer chamber houses the X-ray tube and monochromator.

The monochromator consists of collimators to make the fluorescent X-rays a parallel beam filters to reduce background radiation, crystal to serve as the dispersion medium and detector for measuring the intensities. The sample chamber is separated by means of a movable shutter from the spectrometer chamber. The sample can be introduced into a sample chamber by means of a mechanical arm. The computer and the associated electronic circuits interact with the different parts of the spectrometer to control the sequence of operations and also carry out the mathematical computations to display/print out the final result in terms of percentage.

10.3 Standards and Reagents

10.3.1 *Standards*

Certified reference materials of ferrotitanium of known composition as near in composition as the sample is to be analysed, shall be used for preparing the calibration curve.

10.3.2 *Setting up the Instrument*

Two ferro-titanium standards/ samples, one containing low and the other containing high content of titanium should be used.

10.3.2.1 *Drift Correction*

The intensity from the two standards/samples as described in **10.3.2** may be stored in the computer along with the calibration curve. Before analysing the unknown samples, it is necessary to update the intensities from the standards to take care of any drift in the spectrometer.

10.3.3 *Reagents*

Only high purity anhydrous chemicals such as magnesium oxide, boric acid, lithium tetra borate and lithium iodide should be used.

10.4 Sample Preparation

10.4.1 Samples may be prepared by fusion or by pelletization with binders. Different recipes are available for sample preparation. One method using lithium tetra borate and boric acid is given below.

10.4.2 Weigh 0.2 g of ferrotitanium and evenly spread it over a bed of 1 g magnesium oxide placed inside a platinum lid. Keep it inside a muffle furnace at 5 00°C for 30 min for oxidation. During this period, arrange a free flow of oxygen into the furnace to ensure complete oxidation.

10.4.3 After removing from the furnace allow the lid and its contents to cool in a desiccator. Transfer quantitatively the contents of the lid to a Pt-Rh-Au crucible. Add 8.0 g of anhydrous lithium tetra borate (40 times the weight of the sample), 0.4 g of boric acid and 0.2 g of lithium iodide. Mix the contents of the crucible well and place the crucible in muffle furnace at 1 200°C. Cover the crucible with Pt-Rh-Au lid and allow it to heat for 10 min.

10.4.4 Take out the crucible with a pair of tongs and rotate the crucible for thorough mixing and keep it again in the furnace. After 10 min, remove the lid and place it on an even platform and transfer the fused melt from the crucible to the lid and allow it to cool. When the temperature of the bead comes to that of the surroundings, remove it from the lid. It is now ready for irradiation.

10.5 Calibration and Analysis

10.5.1 Prepare the fused beads of ferrotitanium of certified standard reference materials of known composition following the procedure specified in **10.4**

NOTE — Take care that the ferrotitanium standards selected must be within ± 10 percent of titanium content of the sample to be analyzed.

10.5.2 These beads are then irradiated. The 'K' X-rays of titanium from these beads may be monitored by using a wavelength dispersive, XRF spectrometer described in **10.2.4**.

10.5.3 Select the crystal, detector and the X-ray line listed in Table 1. Each of these beads must be irradiated at least 3 times and the average value of the intensity detected must be used for calibration. These intensities must be correlated to their concentrations and the calibration curve thus obtained may be stored in the computer. The concentration in the case of unknown samples can be obtained by referring to this curve.

Table 1 Selection of Parameters

(*Clause* 10.5.2)

Element	X-ray Line Selected	Wave length *nm*	Crystal	Detector
Titanium	K	2 750	LiF	Any suitable detector

11 DETERMINATION OF TITANIUM CONTENT IN FERROTITANIUM (TITANIUM BETWEEN TWENTY – EIGHTY NINE PERCENT *M/M*) BY — TITRIMETRIC METHOD (ALTERNATE METHOD)

11.1 Outline of the Method

The sample is dissolved in sulphuric, hydrofluoric, nitric and hydrochloric acids. Separation of interfering elements (chromium, vanadium, molybdenum and tin), if present, by precipitation of titanium hydroxide in the presence of hydrogen peroxide. Titanium is reduced to Ti^{3+} by aluminium metal in an atmosphere of carbon dioxide or nitrogen. Ti^{3+} thus obtained is titrated with standard iron (III) ammonium sulphate solution in presence of thiocyanate as indicator.

11.2 Reagents

11.2.1 Aluminium of min. purity 99.5 percent in the form of foil 0.05mm thick and free from titanium

11.2.2 *Sodium Hydrogen Carbonate* ($NaHCO_3$)

11.2.3 *Nitric Acid,* rd = 1.42 (*see* IS 264)

11.2.4 *Sulphuric Acid, rd* = 1.84 (*see* IS 266)

11.2.5 *Hydrochloric Acid,* rd = 1.16 (*see* IS 265)

11.2.6 *Hydrofluoric Acid,* 48 percent

11.2.7 *Sulphuric Acid,* diluted 1 : 1 (*v/v*)

11.2.8 *Sulphuric Acid,* diluted 1 : 4 (*v/v*)

11.2.9 *Sodium Hydroxide Solution,* 100 (*g/ℓ*)

11.2.10 *Sodium Hydroxide Solution,* 20 (*g/*ℓ)

11.2.11 *Ammonium Thiocyanate Solution,* 100 (g/ℓ)

11.2.12 *Nitrogen, Practically Oxygen,* free (<10 ppm by volume), 99.98 percent purity or carbon dioxide of similar purity

11.2.13 *Hydrogen Peroxide Solution,* 30 percent (*v/v*)

11.2.14 *Iron (III) Ammonium Sulphate* $[Fe_2(SO_4)_3.(NH_4)_2SO_4.24\ H_2O]$

Transfer 10.2 g of the Iron (III) ammonium sulphate in a 400 ml beaker and add 100 ml of cold water and 50 ml of the sulphuric acid (*see* **11.2.7**). Transfer the content to a 1 litre volumetric flask and dilute up to the mark with distilled water. 1 ml of this solution corresponds to about 1 mg of titanium.

11.2.15 *Iron (III) Sulphate Solution*

Dissolve 2 *g* of pure iron in 50 ml hydrochloric acid (*see* **11.2.5**), oxidised with 10 ml of the nitric acid (*see* **11.2.3**), add 40 ml sulphuric acid (*see* **11.2.7**) and heat until white fumes are evolved. Cool, dilute to 200 ml and boil for 15 minutes. Cool, transfer quantitatively to a 1 000 ml volumetric flask and make up the volume upto the mark with distilled water.

11.2.16 *Titanium Sponge or Other Metallic Titanium of purity* > 99.9 percent

11.2.17 Titanium, standard solution corresponding to 0.5000 g of titanium per litre, prepared by one of the following methods:

11.2.17.1 *Preparation from titanium dioxide*

11.2.17.1.1 Weigh 0.834 0g ± 0.000 2 g of titanium dioxide which has been previously calcined for 2 h at 800^{O}C. Fuse it gently 10 g of potassium pyrosulphate either in a platinum crucible with lid or in a quartz crucible of capacity 170 ml with a cover, or in a quartz conical flask.

11.2.17.1.2 After cooling, dissolve the fusion product directly in the quartz crucible or in the quartz conical flask with 100 ml of warm water, then decant the solution into 400 ml beaker.

11.2.17.1.3 Rinse the quartz crucible or the quartz conical flask, several times. If a platinum crucible is used, place it, after cooling, in a 400 ml beaker with 100 ml of warm water to dissolve the fusion product. When the product is dissolved, withdraw the crucible, rinsing it several times in the water.

11.2.17.1.4 Add 50 ml of sulphuric acid solution (*see* **11.2.8**) to the solution of the fusion product. Heat gently on a hot plate until the solution becomes completely clear. Cool, transfer quantitatively to a 1 litre volumetric flask, dilute upto the mark with distilled water and mix.

11.2.17.2 *Preparation from potassium hexa flurotitanate*

Weigh 2.506 0g ± 0.000 2 g of potassium hexa flurotitanate (K_2TiF_6) which has been previously dried for 2 h at 105 ^{O}C into a platinum crucible. Place the crucible in a 100 ml beaker, add 50 ml of the sulphuric acid solution (*see* **11.2.8**) and heat until the salt is dissolved (about 15 min). Cool, withdraw the crucible, rinsing it several times with the water. Transfer quantitatively to a 1 litre volumetric flask and volume made upto the mark, after cooling, with distilled water and mix.

11.3 Apparatus

Usual laboratory equipments and in particular the following:

11.3.1 *PTFE beakers of capacity 250 ml;*

11.3.2 *Glass beakers of capacity 500 ml;*

11.3.3 *Conical or round flask with a flat base of capacity 500 ml; and*

11.3.4 *Bunsen valve or goeckel condenser bulb*

The bunsen valve (*see* Fig. 1) comprises a rubber bung (1) through which two glass tubes (2 and 6) pass. Rubber Tubes (3) are fitted on to these glass tubes, the upper ends are being closed by glass plugs (5) the rubber tube (3) which is fitted on the glass tube (2), is slitted (4) for a length 10 mm – 15 mm in the middle of the length by a razor blade. The tube (6) which ends 5 cm above the level of the solution to be titrated makes its possible introduced nitrogen or carbon dioxide by substituting the tube through which the gas is delivered for the rubber tube (3).

11.3.5 *Magnetic Stirrer*, with PTFE covered stirring bar.

11.3.6 Apparatus for titration in an atmosphere of nitrogen (*see* Fig.2).

11.4 Sample

Use sample powder of 160 microns.

11.5 Procedure

11.5.1 *Test Portion*

Take a test portion of 1 ± 0.000 2 g.

11.5.2 *Blank Test*

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents [except the iron (III) ammonium sulphate solution (*see* **11.2.14**)], but omitting the test portion.

11.5.3 *Control Test*

Check the validity of the operating procedures used by carrying out, in parallel with the determination and following the same procedure, the determination(s) of the titanium content(s) of one or several samples of the same type, have been known titanium content(s).

11.5.4 *Standardization of Iron (III) Ammonium Sulphate Solution*

11.5.4.1 *Method using titanium sponge*

Carry out the determination on a test portion of the titanium sponge (*see* **11.2.16**) weighing 0.75 to 0.85 g.

11.5.4.2 *Method using standard titanium solution*

By means of a pipette, transfer to a 500 ml conical flask, 50 ml of the standard titanium solution (*see* **11.2.17**), 5 ml of sulphuric acid solution (*see* **11.2.7**) and 80 ml of hydrochloric acid (*see* **11.2.5**).

Proceed as specified in **11.5.5.3.2.**

Carry out the blank test under the same conditions in order to verify the absence of titanium in the reagents used for preparation of the standard solution.

11.5.5 *Determination*

11.5.5.1 *Dissolution of the sample*

11.5.5.1.1 Place the test portion in a 250 ml PTFE beaker, and add about 20 ml of water and 35 ml of

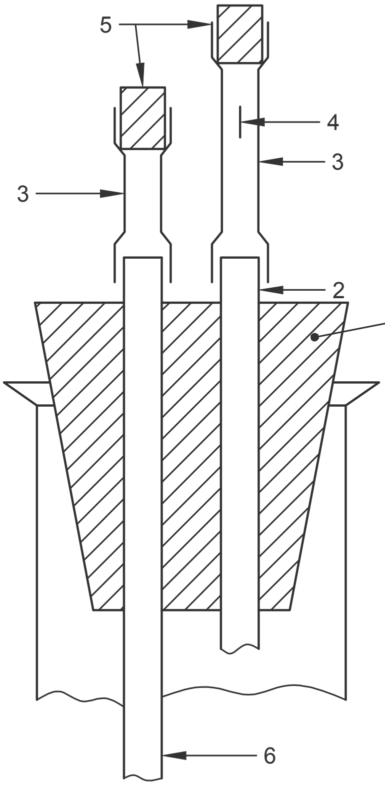

Fig. 1 Bunsen Valve

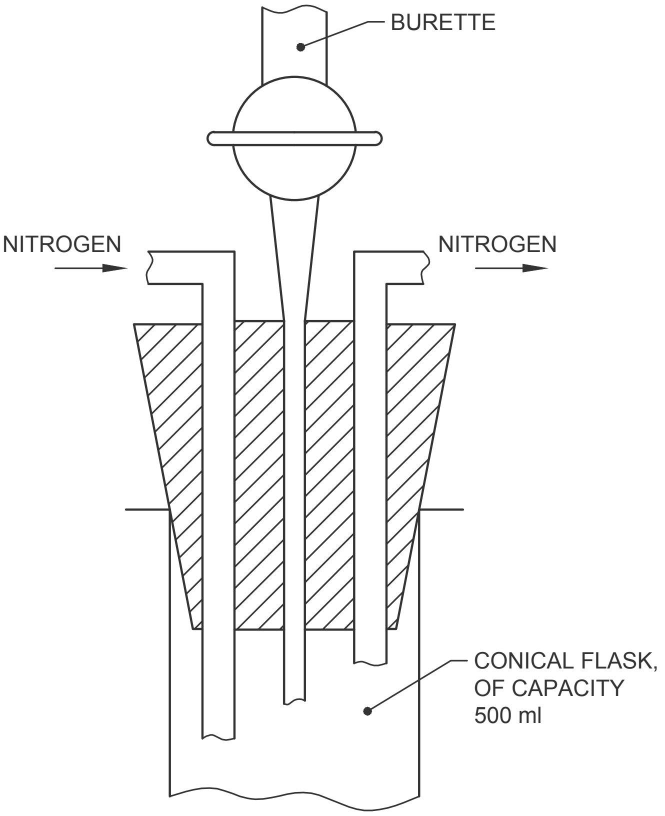

Fig. 2 Apparatus for Titration in an Atmosphere of Nitrogen

sulphuric acid solution (*see* **11.2.7**). Then the reaction has slowed, add 20 ml of the hydrochloric acid (*see* **11.2.5**) and 1 ml of hydrofluoric acid (*see* **11.2.6**). Cover the beaker with a PTFE cover. Oxidized while cold by adding, drop by drop 5 ml of nitric acid (*see* **11.2.3**).

11.5.5.1.2 When the reaction is complete, remove the cover and heat gently until white fumes are evolved. Continue heating for about 5 min. Cool, add 30 ml of the hydrochloric acid (*see* **11.2.5**), stir gently, cover and heat on a hot-plate until the solution becomes clear. Rinse the cover with 5 ml of the hydrochloric acid (*see* **11.2.5**).

11.5.5.1.3 Cool and transfer the solution to a 500 ml volumetric flask, rinse the beaker and dilute the volume upto the mark with distilled water (solution *A*).

11.5.5.1.4 If the expected titanium content of the sample lies in the range 20 – 45 percent (*m/m*) take exactly 50 ml of solution '*A*', if the expected titanium content lies in the range 45 – 75 percent (m/m), take exactly 25 ml of the solution '*A*'.

11.5.5.1.5 Transfer this aliquot portion to a 250 ml beaker containing 10 ml of the iron (III) sulphate solution (*see* **11.2.15**) if the interfering elements are to be separated, proceeding as specified in **11.5.5.2**, or into a 500 ml conical flask if this separation is not to be carried out, proceeding as specified in **11.5.5.3.2**.

11.5.5.2 *Separation of interfering elements*

11.5.5.2.1 Add to the aliquot portion of solution *'A'* in the 250 ml beaker, 50 ml of the sodium hydroxide solution (*see* **11.2.9**) and 2 ml of the hydrogen peroxide solution (*see* **11.2.13**), heat to boiling and keep boiling for 5 min.

11.5.5.2.2 Allow the precipitate to settle and filter either through a pulp pad. After having transferred all the hydroxide precipitate on to the filter, wash the filter assembly and the precipitate with the sodium hydroxide solution (*see* **11.2.10**) and rinse the beaker and precipitate 6 times with about 10 ml of the sodium hydroxide solution (*see* **11.2.10**). Rinse the stem of the funnel with water and place the funnel on a 500 ml conical flask. Take 45 ml of hydrochloric acid (*see* **11.2.5**) and 15 ml of the sulphuric acid solution (*see* **11.2.8**) In a separate 250 ml beaker and heat to 60–70^{O}C. Using this acid mixture, dissolve the hydroxide precipitate on the filter, adding the mixture in fractions of 10 ml and allowing each fraction to run away completely before repeating the addition. When the beaker is empty, add 35 ml of the hydrochloric acid (*see* **11.2.5**) heat as before, and wash the filter with fractions of about 10 ml of the hydrochloric acid. Finally rinse the beaker, the filter and the stem of the funnel with hot water, using a total volume not exceeding 40 ml. Proceed as specified in **11.5.5.3.2**.

11.5.5.3 *Titration*

11.5.5.3.1 *Absence of interfering elements*

Add to the aliquot portion of solution 'A' in the 500 ml conical flask, 85 ml of the hydrochloric acid (*see* **11.2.5**) and 25 ml of water.

Proceed as specified in **11.5.5.3.2**

11.5.5.3.2 *Reduction of titanium*

Add to the test solution (prepared in **11.5.5.2** or **11.5.5.3.1**) to ± 0.2 g of the sodium hydrogen carbonate (**11.2.2**) and 4 ± 0.2 g of the aluminium (*see* **11.2.1**), cut into several pieces. Immediately stopper the flask with the bunsen valve (*see* **11.3.4**) filled with saturated sodium hydrogen carbonate solution. Agitate very frequently during the reduction.

NOTE — At the end of the reduction, suction may be produced in the bunsen valve and it will then be necessary to add the saturated sodium hydrogen carbonate solution rapidly.

11.5.5.3.3 *Titration in an atmosphere of nitrogen*

Before the aluminium is totally dissolved, connect the descending (6) of the bunsen valve to the nitrogen source after having withdrawn the rubber (3). Adjust the nitrogen flow to 0.7 ± 0.1 litres/min. When the aluminium is dissolved (cessation of effervescence), immerse the flask totally in cold water and allow the nitrogen to escape through the slit (4) in the rubber tube (3) and leave until the solution is at ambient temperature (about 7 minutes is required).

Without interrupting the flow of nitrogen lift the bunsen valve and add 10 ml of ammonium thiocyanate solution (*see* **11.2.11**) and the bar of the magnetic stirrer (*see* **11.3.3**) rinse the descending tube (6) with cold boiled water and replace the bunsen valve with the titration assembly (*see* Fig. 2), after having connected it to the source of nitrogen. Place on the magnetic stirrer (*see* **11.3.5**) and titrate with the iron (III) ammonium sulphate solution (*see* **11.2.14**) until a persistent pink colour is obtained.

11.5.5.3.4 *Titration in a atmosphere of carbon dioxide*

When the aluminium is dissolved, cool the solution and withdraw the bunsen valve. Add 10 ml of the ammonium thiocyanate solution (*see* **11.2.11)** and titrate with the iron (III) ammonium sulphate solution (*see* **11.2.14**) until a persistent pink colour is obtained.

11.6 Expression of Results

11.6.1 If the iron (III) ammonium sulphate solution was standardised by the method using titanium sponge (*see* **11.5.4.1**), the titanium content, is expressed as percentage by mass, is given by the formula:

$$\frac{m_1}{V_2 - V_1} \times \frac{V_3 - V_1}{m_2} \times 100$$

where,

V_1 = volume of iron (III) ammonium sulphate solution used in the blank test, in ml;

V_2 = volume of iron (III) ammonium sulphate solution used for the standardizing titanium sponge, in ml;

V_3 = volume of iron (III) ammonium sulphate solution used in the determination, in ml;

m_1 = mass of titanium sponge (*see* **11.2.16**) contained in the aliquot portion taken in **11.5.5.1**, in g; and

m_2 = mass of ferrotitanium contained in the aliquot portion taken in **11.5.5.1,** in g**.**

11.6.2 If the iron (III) ammonium sulphate solution was standardized by the method using standard titanium solution (*see* **11.5.4.2**), the titanium content, is expressed as percentage by mass, is given by the formula:

$$\frac{m_1}{V_2 - V_1} \times \frac{V_3 - V_1}{m_2} \times 100$$

where,

V_1 = volume of iron (III) ammonium sulphate solution (*see* **11.2.14**) used in the blank test, in ml;

V_2 = volume of iron (III) ammonium sulphate solution used to titrate 50 ml of the standard titanium solution, in ml;

V_3 = volume of iron (III) ammonium sulphate solution used in the determination, in ml;

V_4 = volume of iron (III) ammonium sulphate solution used in the blank test, in ml;

m_1 = mass of titanium contained in 50 ml of standard titanium solution, in g; and

m_2 = mass of ferrotitanium contained in the aliquot portion taken in **11.5.5.1,** in g.

11.7 Test Report

The test report shall include the following particulars:

a) Identification of the sample, and
b) The result and the method of expression used.

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This Indian Standard has been developed from Doc No.: MTD 34 (5220).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi